FINAL

US Army Corps of Engineers

Toxic and Hazardous Materials Agency

APPENDED QUALITY ASSURANCE PROJECT PLAN FOR TASK ORDER 1

for the

RCRA Facility Investigation/Corrective Measures Study (RFI/CMS) and Base Closure Environmental Study for the Lexington-Blue Grass Army Depot

Submitted to:

Commander
Department of the Army
United States Army Toxic and Hazardous Material Agency
Aberdeen Proving Ground, Maryland

Submitted by

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Prepared Under:

Contract No. DAAA15-90-D-0016
Task Order Number 4

October 24, 1991

THAMA Form 45, 1 Jul 90

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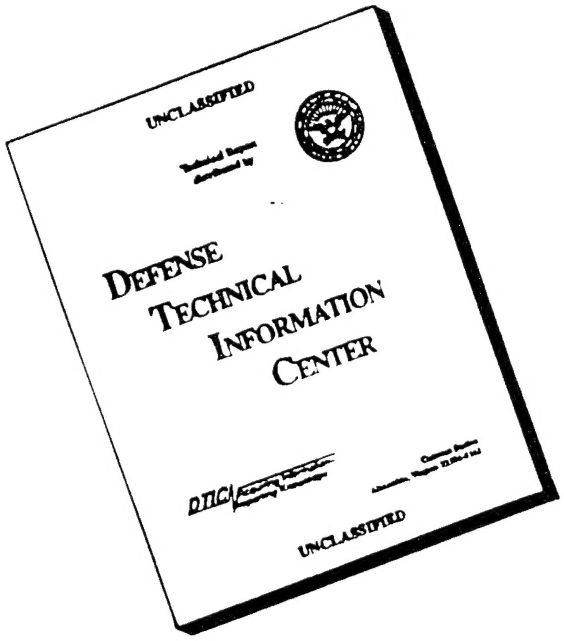

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INTRODUCTION

Metcalf & Eddy, Inc. (M&E) under contract to the United States Army Toxic and Hazardous Materials Agency (USATHAMA), contract number DAAA15-90-D-0016, Task Order Number 4, has appended the plans prepared for the RFI/CMS base closure at the Lexington-Blue Grass Army Depot, Kentucky. This document is one of these appendices -- the appendix to the Quality Assurance Project Plan. The appended document, Task Order 1, Quality Assurance Project Plan (QAPP), Lexington-Blue Grass Army Depot, Kentucky (USATHAMA, 1991) is henceforth called the "original document."

USEPA review comments of July 31, 1991, on the original document were incorporated into this appended document unless the comment requested work outside the task order #4 scope of work. Work outside the scope will be conducted at a later date, as deemed necessary by the COR.

AMENDMENTS/CLARIFICATIONS/ADDITIONS TO THE QAPP

The QAPP is hereby amended/clarified/appended as follows.

Section 1.0 through 4.0, Pages i through 8. These pages of the original plan are unchanged and apply as written.

Section 5.0, Page 9, 6th bullet.

Change: Delete entire bullet.

Section 5.0, Page 9, 7th bullet.

Change: Delete entire bullet.

Section 5.1.1, Page 9, 1st Paragraph, 1st Sentence.

Change: After "...groundwater,"

Change to: "wipe samples."

Section 5.1.1, Page 9, 1st Paragraph, 2nd Sentence.

Change: "Specific methods have been selected based upon laboratory certifications within the USATHAMA program."

Change to: "Methods used for analyses will be certified by USATHAMA, if required. "USATHAMA-Certified" methods are based on EPA methods and are listed below."

Section 5.1.1, Page 9, 1st Paragraph, 1st Sentence.

Change: "...in Appendix B to this document"

Change to: "...in Appendix A to this document"

Section 5.1.1.1, Page 10, 1st Paragraph, Last Sentence.

Change: Delete "are presented in Appendix B."

Change to: "are those associated with the certified methods listed in Appendix A."

Section 5.1.1.1, Page 10, 2nd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The anticipated quantification limits are the certified reporting limits associated with the USATHAMA certified methods listed in Appendix A."

Section 5.1.1.1, Page 10, 3rd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The anticipated detection limits are the certified reporting limits associated with the USATHAMA certified methods listed in Appendix A."

Section 5.1.1.2, Page 10, 1st Paragraph, Last Sentence.

Change: Delete "are presented in Appendix B."

Change to: "are those associated with the certified methods presented in Appendix A."

Section 5.1.1.2, Page 10, 2nd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The detection limits are the certified reported limits associated with the method and are listed in Appendix A."

Section 5.1.1.2, Page 10, 4th Paragraph, Last Paragraph.

Change: Delete "Appendix B lists the anticipated quantization range..."

Change to: "Appendix A lists the certified reporting limits..."

Section 5.1.1.3, Page 10, 2nd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The certified report limits are listed for each analyte by matrix in Appendix A."

Section 5.1.1.4, Page 11, 3rd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The CRLs are listed in Appendix A."

Section 5.1.1.5, Page 11, 3rd Paragraph, Last Sentence.

Change: Delete last sentence.

Change to: "The CRLs for Hg analysis is listed in Appendix A."

Section 5.1.1.6, Page 11, 3rd Paragraph.

Change: Delete 3rd Paragraph.

Section 5.1.1.7, Page 11, 1st Paragraph, Last Sentence.

Change: Delete last sentence.

Section 5.1.1.8, Page 11, 1st Paragraph, Last Sentence.

Change: Delete "Appendix B"

Change to: "Appendix A"

Section 5.1.1.8, Page 11, 2nd Paragraph, 1st Sentence.

Change: Delete "listed in Appendix B"

Change to: "the CRLs listed in Appendix A"

Sections 5.1.1.9 through 5.1.1.11. These pages of the original plan are unchanged and apply as written.

Section 5.1.1.12, Page 12.

Change: Delete entire section.

Section 5.1.1.13, Page 12, 1st Paragraph, 1st Sentence.

Change: Add "and soil samples" after "water".

Section 5.1.1.13, Page 12, 1st Paragraph, 2nd Sentence.

Change: Delete last sentence.

Section 5.1.1.13, Page 12, 2nd Paragraph.

Change: Delete last sentence.

Change to: "Quantitation limits will comply with local Underground Storage Tank regulations."

Section 5.1.1.14, Page 12, 2nd Paragraph.

Change: Delete 2nd paragraph.

Section 5.2, Page 12, 1st Paragraph.

Change: After second sentence, add: "Precision for USATHAMA certified methods are measured through the use of control charts."

Section 5.2, Page 12, 3rd Sentence.

Change: Add "of non-certified methods" after "The precision."

Section 5.3, Page 14, 1st Paragraph, Last Sentence.

Change: Delete last sentence.

Section 5.3, Page 14, 1st Paragraph, 5th Sentence.

Change: Add "wipe" after "soils."

Section 5.4, Page 14, 2nd Paragraph, Last Sentence.

Change: Delete "for both soil and water matrices"

Change to: "for soil, wipe, and water matrices."

Sections 5.5 and 5.6. These sections of the original plan are unchanged and apply as written.

Section 6.1, 6.2, 6.3, 6.4, 6.5, 6.6, 6.7, 6.8. These sections of the original plan are unchanged and apply as written.

Section 6.9, Page 20.

Change: Delete existing paragraphs.

Change to: Assigning sample identification numbers is described in Section 3.6 of the Technical Plan.

Sections 7.0 through 7.3. These sections of the original plan are unchanged and apply as written.

Section 9.4, Page 24.

Change: Delete all but last sentence.

Sections 9.5 through 10.7.1. These sections of the original plan are unchanged and apply as written.

Section 10.8, Page 27, 1st Paragraph, 1st Sentence.

Change: Delete 1st sentence.

Change to: "Data packages will be thoroughly reviewed by the laboratory QA Coordinator prior to submission to the USATHAMA Data Management system."

Sections 11.0 through the end (excluding tables, etc.). These sections of the original plan are unchanged and apply as written.

Table S-1.

Change: Delete all references to TCLP.

Change to: Reference TCL.

Table 6-1.

Change: Under preservatives for cyanide in water, add: "0.6 Ascorbic acid if residual chlorine is present."

Table 10-1. This table is unchanged and applies as written.

Figures. This section is unchanged and applies as written.

Appendix A and B.

Change: Delete Appendices A and B.

Change to: Insert Data Chem QAPP as Appendix A.

To Appendix B - Data Chem QAPP.

Change: Delete Appendix B of Data Chem QAPP.

Change to: Table 6-1 (as amended).

QUALITY ASSURANCE PROGRAM PLAN

for
U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY

Laboratory Analysis
of Environmental Samples

DCL Document QA-3/87
August 1991

DataChem Laboratories
960 West LeVoy Drive
Salt Lake City, Utah 84123

TABLE OF CONTENTS

1.0
DOCUMENT IDENTIFICATION

Document Title:	Quality Assurance Program Plan for USATHAMA
Document Control Number:	QA-3/87
Organization:	DataChem Laboratories (DCL) 960 W. LeVoy Dr. Salt Lake City, Utah 84123
Director:	James H. Nelson, Ph.D. Phone: 801-266-7700
Quality Assurance:	Ronald H. Marsden, B.S. Phone: 801-266-7700

2.0
INTRODUCTION

This document is the DCL Quality Assurance/Quality Control Plan, prepared in compliance with the requirements of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) with analytical laboratory services in support of the implementation of various installation restoration programs. This plan adheres to, and is an implementation of, the USATHAMA QA Program, January 1990, First Edition.

DCL is committed, in strictly following this plan, to provide to USATHAMA analytical data that are of a quality that may be used in litigation. All deviations from this plan or the USATHAMA QA Program will be submitted to USATHAMA for approval prior to implementation in the laboratory. Such deviations will be properly and fully documented.

DCL has conducted analyses for USATHAMA since 1984 under the 1982 USATHAMA QA Program, the Second Edition (March 1987) of the 1985 USATHAMA QA Program, and the January 1990 USATHAMA QA Program, First Edition.

3.0
ORGANIZATION AND RESPONSIBILITIES

3.1 Introduction

Ultimate responsibility for the conduct of all projects, and approval for the implementation of all programs at DCL resides with the Laboratory Director, Dr. James H. Nelson. Functional responsibility for the analytical work is delegated to the Project Manager, Mr. David W. Gayer; to the Analytical Task Managers, Mr. A. Brent Torgensen, and Mr. Richard Wade; and to the Quality Assurance Coordinator, Mr. Ronald H. Marsden.

3.2 Laboratory Director

The Laboratory Director is responsible to assure that DCL resources are adequately allocated to the project and that sufficient staffing and equipment are provided. He oversees and supports the Quality Assurance Coordinator.

3.3 Project Manager

The Project Manager has the responsibility of communication with the USATHAMA Program Contract Officer and oversees and supports the Analytical Task Managers in development, implementation, and operation of the analytical program organization. He is directly responsible for the interpretation of the provisions of the contract for DCL. The Project Manager is also responsible to assure that QA/QC recommendations and corrective actions are implemented.

The Project Manager is authorized to conduct official discussions with the Program Contract Officer concerning the original contractual agreement and delivery orders, and any subsequent modifications to the contractual agreement and/or delivery orders. Laboratory personnel matters are decided in concert with the Analytical Task Manager and appropriate Section Managers.

3.4 Analytical Task Manager

The Analytical Task Manager has the responsibility of implementing the USATHAMA 1990 QA Plan, and for coordinating the sample analysis flow in the laboratory. This will be achieved through the following:

1. Assuring the provision of sufficient equipment, laboratory space, resources, personnel, and quality reagents and materials to properly conduct the required analyses;

2. Supporting the Quality Assurance Coordinator;

3. Submitting documented analytical methods and laboratory certification data to the USATHAMA Project Officer prior to the analysis of field samples;

4. Ensuring that all provisions of the approved Project Quality Control Plan are fully implemented in the laboratory;

5. Ensuring the implementation of corrective action for any QA/QC deficiencies.

The Analytical Task Manager has the authority to suspend analytical work for quality control problems and to implement corrective actions recommended by the Quality Assurance Coordinator. He also has authority to accept or reject increases in the delivery rate of samples, within the bounds set by the contract. He confers with section managers and the Project Manager on personnel matters when they impact on the project.

3.5 Quality Assurance Coordinator

The Quality Assurance Coordinator (QAC) has the responsibility of establishing, overseeing, and auditing specific procedures for documenting, controlling, and validating analytical data quality. This is accomplished, in part, through the following:

1. Monitoring the QA and QC activities of the laboratory to ensure conformance with authorized policies, procedures, and good laboratory practices, and recommending improvements as necessary;

2. Informing the Project Manager and/or the Analytical Task Manager of noncompliance with the approved QA Program;

3. Requesting standard analytical reference materials from USATHAMA;

4. Ensuring that all records, logs, standard operating procedures, project plans and analytical results are maintained in a retrievable fashion;

5. Ensuring that standard operating procedures and project QA/QC plans are distributed to all appropriate laboratory personnel;

6. In consultation with the analysts and the Analytical Task Manager, establishing appropriate analytical lot size, including the correct QC samples;

7. Establishing the correct procedures and criteria for evaluating whether analytical performance is acceptable and in-control;

8. Ensuring that samples are received and logged properly, including lot sizing, introduction of required QC samples, and numbering of field samples and control samples;

9. Reviewing all laboratory data before those data are released, verifying that data were collected properly under an in-control analytical system;

10. Ensuring that the DCL quality control chemist, or appropriate analysts, are properly preparing QC samples;

11. Maintaining quality control charts, ensuring timely distribution of such charts, documenting corrective actions, and ensuring that analysts implement and document corrective actions as they become necessary;

12. Ensuring that sample logs, instrumentation logs, and all QC documents are properly maintained, including frequency of entries;

13. Discussing control chart results with the Analytical Task Manager and submitting updated, current charts to the USATHAMA Project Officer on a weekly basis, or as required by USATHAMA;

14. Maintaining an awareness of the entire laboratory operation to detect conditions which might jeopardize controls of the various analytical systems;

15. As directed by USATHAMA, auditing sampling documentation and procedures to ensure proper labeling, handling, transportation, and storage.

The Quality Assurance Coordinator has the authority to:

1. Approve all analytical reports;

2. Reject analytical data which does not meet applicable quality control criteria;

3. Require re-performance of sample analyses which are determined to be out-of-control;

4. Evaluate data and determine apparent long-term trends which may require corrective action;

5. Suspend analytical work, when necessary, to assure corrective actions are taken and that an analysis is again in control.

The Quality Assurance Coordinator also attends and participates in conferences for discussion of quality control and quality assurance problems and procedures.

4.0
CERTIFICATION

4.1 Laboratory Certification

DCL, as a laboratory, rather than as individual analysts, certifies as proficient in conducting analyses for USATHAMA. Each member of the organization has the education and training necessary to enable that individual to perform assigned functions. A personnel training file is maintained for each individual. Each individual updates the training file as necessary.

Management personnel have earned a Baccalaureate degree from an accredited college or university.

Analytical Chemists have earned a Baccalaureate Degree in Science or related fields from an accredited college or university.

Technical Staff have applicable training, including on the job training, and/or experience in related fields.

4.2 Analytical Methods

Analytical methods used for the analysis of environmental samples are described in a set of written instructions completely defining the procedure to be followed to process a sample and obtain an analytical result. An analytical method describes, as a minimum, the analytes for which it is valid, the matrix type, sample preparation, reagent and standards preparation, instrument calibration, and computations used to evaluate the analytical results. Standards and quality control sample requirements are also defined.

Analytical methods are either supplied by USATHAMA or, with approval, developed by DCL. The documentation for proposed methods development includes:

1. The submission of documentation to USATHAMA.
2. A statement of the problem.
3. A description of the technical approach to include specific details on procedures, solvents, instrumentation, etc.
4. An estimate of resources required (to include labor hours, funds and schedule).

When the testing of the analytical procedures has been successfully completed, the method is documented in the standardized USATHAMA format. The format for documentation of all analytical methods is provided in Table 1. The format for data analysis is established by USATHAMA-provided statistical analysis computer software. Updates to the software are implemented upon receipt.

Table 1.
FORMAT FOR DOCUMENTATION OF METHOD CERTIFICATION

I. Summary
- A. Analytes
- B. Matrix
- C. General Method

II. Application
- A. Tested Concentration Range
- B. Sensitivity
- C. Reporting Limit
- D. Interferences
- E. Analysis Rate
- F. Safety Information

III. Glassware and Chemicals
- A. Glassware/Hardware
- B. Instrumentation
- C. Analytes
- D. Reagents and SARMs

IV. Calibration
- A. Initial Calibration
- B. Daily Calibration

V. Certification Testing

VI. Sample Handling and Storage
- A. Sampling Procedure
- B. Containers
- C. Storage Conditions
- D. Holding Time Limits
- E. Solution Verification

VII. Procedure
- A. Separations
- B. Chemical Reactions
- C. Instrumental Analysis
- D. Confirmational Analysis

VIII. Calculations

IX. Daily Quality Control
- A. Control Samples
- B. Control Charts

X. References

XI. Data

The analytical method, once certified, is followed for all USATHAMA analyses. Any permitted deviations, such as oven temperature or gas flow rate, are documented in the analyst's notebook and the data file. Permanent changes in a certified method are submitted to USATHAMA for approval prior to implementation.

All copies of USATHAMA-certified methods are individually numbered. Each distributed method copy must be signed for and dated. A comprehensive list of all distributed methods is kept by the Quality Assurance Coordinator.

4.3 Method Certification

Before field samples may by analyzed by the laboratory, the methods of analysis must be certified. Certification for selected methods, accomplished under other USATHAMA contracts, may be determined by USATHAMA to be acceptable for the work performed under this contract for identical analytes and matrices. If analytes are required for a particular certified method in addition to those which have already been certified, the additional analytes are appended to the current certified method by following full certification procedures for the additional analytes. The current certified method standards, concentrations and analytical conditions are used to certify the additional compounds.

Some methods, including calibration of test and measurement equipment, do not require certification, due to either the nature of the measurement or the intended use of the data. When such methods are part of a project, USATHAMA will not provide a standardized method. However, laboratories must submit sufficient information in test plans, work plans, and project QC plans to describe exactly the procedures to be used. A copy of a proposed method must be submitted to the USATHAMA Chemistry Branch before it is used on any project.

The following methods do not require USATHAMA certification by the USATHAMA Chemistry Branch: temperature, conductivity, pH, oil and grease, hardness, asbestos, alkalinity (carbonate/bicarbonate/hydroxide), total organic carbon, biochemical oxygen demand, chemical oxygen demand, total dissolved solids, total suspended solids, totals solids, total petroleum hydrocarbons, salinity, and acidity.

4.3.1 Written Method

A draft of the analytical method proposed for certification is submitted to USATHAMA for approval with the precertification performance data package.

4.3.2 Standards

Standard Analytical Reference Materials (SARMs), provided by USATHAMA, are used in all method certification analyses. DCL obtains suitable, certified Reference Materials from the EPA or other commercial sources for analytes for which USATHAMA is not able to provide SARMs. Standard water and standard soil are used by DCL for all USATHAMA analyses done.

4.3.3 Standard Water

Standard water samples are prepared by adding a known quantity of target analyte to a known volume of water. The volume of water is specific in the method being performed. All target analytes for the method are added. ASTM Type I grade water is used for inorganic methods. ASTM Type II grade water containing 100 mg/L each of added sulfate and chloride is used for organic methods. The method and reagents used to prepare spiking solutions are specified in the standardized methods.

4.3.4 Standard Soil

Standard soil samples are prepared by adding a known quantity of target analyte to a known weight of selectively blended standard soil as provided by the Chemistry Branch of USATHAMA.

4.3.5 Precertification Calibration

Before initiating method certification, precertification calibration is performed. DCL holds discussions with USATHAMA delineating anticipated environmental concentrations. The concentration range tested includes the Target Reporting Limit (TRL). Additional concentrations of calibration standards may be included for expanding the range of certification. Duplicate analyses are performed on all of the calibration standards.

The certified check standards are obtained from a source other than USATHAMA, whenever possible. In the absence of suitable commercially prepared mixtures, the DCL Quality Control Chemist prepares appropriate mixtures from certified pure stock reagents. The mixtures contain the analyte(s) of interest at concentrations near the high end of the certification range.

The calibration standard data is tabulated and graphed for analysis of Lack of Fit (LOF) and Zero Intercept (ZI), then submitted to USATHAMA for evaluation. The check standard results are required to fall within the acceptability limits defined by the originator.

4.3.6 Certification

Certified methods meet the following conditions: The Target Reporting Limit (TRL) and the range of certification are selected in consultation with USATHAMA. A pre- certification analysis is performed and reported to USATHAMA, with a copy of the analytical method. Upon approval from USATHAMA, a Class 1, Class 1A, Class 1B, or Class 2 certification process is initiated. See Table 2.

Data derived from certification is processed using USATHAMA supplied software, and submitted to USATHAMA for evaluation. The method Certified Reporting Limit (CRL) and certified range are determined from this data evaluation.

Methods certified under previous editions of the USATHAMA Quality Assurance Program and determined by USATHAMA to be valid for current work do not require recertification.

All certification data are properly maintained in archive files.

4.3.7 Method Modifications and Control

Any modifications, additions, or deletions proposed to any USATHAMA-certified method must be submitted to USATHAMA for approval before such a change is made. Following approval, the revised method (with changes plainly noted) shall be distributed to appropriate laboratory personnel as described in DCL SOP-GLP-002, and the old method collected for retirement.

4.4 Analyst Training

An analyst certifying a new method is qualified to perform that method during routine field sample analysis. An analyst who is required to perform on a procedure which has already been certified is required to satisfactorily analyze an appropriate set of quality control samples to demonstrate ability to perform the method. The demonstration sample data must pass current quality control criteria. Successful certification performance is reflected by an addition to the analyst's training file.

The analyst prepares all data records and a data package, as required for field sample analysis data. The data and the data package must be approved by Quality Assurance. The data and data package are maintained in archives.

Table 2.
NUMBERS AND CONCENTRATIONS OF CALIBRATION STANDARDS (LINEAR AND ZERO-INTERCEPT)

PRECERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 12 Standards + 1 Check Standard (SC)
Blank, *0.5, 1, 2, 5, & *10 TRL (Duplicate) + CS
MTR + 1 Order of Magnitude Extension: 18 Standards + 1 Check Standard (CS)
Blank, *0.5, 1, 2, 5, 10, 20, 50, & *100 TRL (Duplicate) + CS
MTR + 2 Orders of Magnitude Extension: 24 Standards + 1 Check Standard (CS)
Blank, *0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, & *1000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 8 Standards
Blank, *0.5, 2, & *10 TRL (Duplicate)
MTR + 1 Order of Magnitude Extension: 12 Standards
Blank, *0.5, 2, 10, 50, & *200 TRL (Duplicate)
MTR + 2 Orders of Magnitude Extension: 16 Standards
Blank, *0.5, 2, 10, 50, 200, 500, & *2000 TRL (Duplicate)

PRECERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 8 Standards + 1 Check Standard (CS)
Blank, *0.5, 2, & *10 TRL (Duplicate) + CS
MTR + 1 Order of Magnitude Extension: 12 Standards + 1 Check Standard (CS)
Blank, *0.5, 2, 10, 50, & *200 TRL (Duplicate) + CS
MTR + 2 Orders of Magnitude Extension: 16 Standards + 1 Check Standard (CS)
Blank, *0.5, 2, 10, 50, 200, 500, & *2000 TRL (Duplicate) + CS

PRECERTIFICATION - CLASS 2

(Not Required)

INITIAL CALIBRATION - CLASS 1

Minimum Testing Range (MTR): 7 Standards + 1 Check Standard (CS)
Blank, *0.5, 1, 2, 5, *10, & *10 TRL + CS
MTR + 1 Order of Magnitude Extension: 10 Standards + 1 Check Standard
Blank, *0.5, 1, 2, 5, 10, 20, 50, *100, & *100 TRL + CS
MTR + 2 Orders of Magnitude Extension: 13 Standards + 1 Check Standard
Blank, *0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, *1000, & *1000 TRL + CS

* 10 percent to 25 percent Range Extension

Table 2
(Continued)

INITIAL CALIBRATION - CLASS 1A

Minimum Testing Range (MTR): 5 Standards
Blank, *0.5, 2, *10, & *10 TRL
MTR + 1 Order of Magnitude Extension: 7 Standards
Blank, *0.5, 2, 10, 50, *200, & *200 TRL
MTR + 2 Orders of Magnitude Extension: 9 Standards
Blank, *0.5, 2, 10, 50, 200, 500, *2000, & *2000 TRL

INITIAL CALIBRATION - CLASS 1B

Minimum Testing Range (MTR): 5 Standards + 1 Check Standard (CS)
Blank, *0.5, 2, *10, & *10 TRL + CS
MTR + 1 Order of Magnitude Extension: 7 Standards + 1 Check Standard
Blank, *0.5, 2, 10, 50, *200, & *200 TRL + CS
MTR + 2 Orders of Magnitude Extension: 9 Standards + 1 Check Standard
Blank, *0.5, 2, 10, 50, 200, 500, *2000, & *2000 TRL + CS

INITIAL CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 6 Standards
Blank and 1 TRL (Triplicate)

DAILY CALIBRATION - CLASS 1/CLASS 1A/ CLASS 1B

Minimum Testing Range (MTR): 2 Standards
*10 & *10 TRL
MTR + 1 Order of Magnitude Extension: 2 Standards
*100 & *100 TRL
MTR + 2 Orders of Magnitude Extension: 2 Standards
*1000 & *1000 TRL

DAILY CALIBRATION - CLASS 2

Minimum Testing Range (MTR): 4 Standards
Blank and 1 TRL (Duplicate)

Table 2
(Continued)

CERTIFICATION - CLASS 1

Minimum Testing Range (MTR): 9 Initial, 6 Daily
MTR + 1 Order of Magnitude Extension: 12 Initial, 6 Daily
MTR + 2 Orders of Magnitude Extension: 15 Initial, 6 Daily

CERTIFICATION - CLASS 1A

Minimum Testing Range (MTR): 5 Initial
MTR + 1 Order of Magnitude Extension: 7 Initial
MTR + 2 Orders of Magnitude Extension: 9 Initial

CERTIFICATION - CLASS 1B

Minimum Testing Range (MTR): 6 Initial, 6 Daily
MTR + 1 Order of Magnitude Extension: 8 Initial, 6 Daily
MTR + 2 Orders of Magnitude Extension: 10 Initial, 6 Daily

CERTIFICATION - CLASS 2

Minimum Testing Range (MTR): 6 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1

Minimum Testing Range (MTR): 9 Initial
MTR + 1 Order of Magnitude Extension: 12 Initial
MTR + 2 Orders of Magnitude Extension: 15 Initial

INITIAL FIELD SAMPLE LOT - CLASS 1A

Minimum Testing Range (MTR): 5 Initial
MTR + 1 Order of Magnitude Extension: 7 Initial
MTR + 2 Orders of Magnitude Extension: 9 Initial

Table 2
(Continued)

INITIAL FIELD SAMPLE LOT - CLASS 1B

Minimum Testing Range (MTR): 6 Initial
MTR + 1 Order of Magnitude Extension: 8 Initial
MTR + 2 Orders of Magnitude Extension: 10 Initial

INITIAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 6 Initial

ADDITIONAL FIELD SAMPLE LOT - CLASS 1/CLASS 1A/CLASS 1B

Minimum Testing Range (MTR): 2 Daily
MTR + 1 Order of Magnitude Extension: 2 Daily
MTR + 2 Orders of Magnitude Extension: 2 Daily

ADDITIONAL FIELD SAMPLE LOT - CLASS 2

Minimum Testing Range (MTR): 4 Daily

5.0
SAMPLE HANDLING AND ANALYSIS

5.1 Sample Management

In most instances, DCL does not perform sample collection, but receives samples from designated field crews. Samples received by DCL are received by designated sample custodians. The protocols of sample management are delineated below.

5.1.1 Sample Containers

As directed by USATHAMA, DCL will supply sample bottles and/or shipping coolers for use in the collection of field samples. A copy of DCL's "Field Sampling Information," to be used as guidance in sampling and in the completion of chains-of-custody, is included in the initial shipment of coolers to the field sampling site. All sample containers shall be cleaned before use according to the protocols specified in Appendix C. Use of commercially cleaned bottles is acceptable provided that cleaning is performed as specified in Appendix C or meets the requirements of the EPA's Contract Laboratory Program.

Generally, for water samples, this includes: septum-sealed glass vials for volatile compounds; amber glass bottles with Teflon-lined lids for organic constituents other than volatiles; and polyethylene bottles for inorganic analytes. Exceptions are noted in the certified method. For soil and sediment samples wide-mouth amber-glass bottles shall be used. Preservatives, as delineated in the DCL USATHAMA Analyte Summary (Appendix B), are provided (as necessary) with sample containers shipped to the field, for proper addition at the site.

5.1.2 Sample Receipt

Samples are received at DCL by the designated Sample Receipt Officer (SRO), or his designee. At the time of receipt of a sample shipment, the sample shipping containers are opened and the samples are inspected. A Sample Receipt Form is initiated at this time. This form includes entries for date and time of receipt, airbill number, a record of the condition of seals on the shipping container and samples, documentation present, temperature and general condition of the shipment, and correlation of sample document and sample labeling information.

Any discrepancies between the samples and the documentation, including missing, broken, or damaged samples, will be reported to USATHAMA or its contractor within 24 hours.

The SRO or his designee signs the field chain-of-custody record at the time that the shipping container is opened. In the case of water samples, which do not usually require splitting, the SRO or his designee opens the shipping container and completes the sample inspection form and field chain-of-custody record. Sufficient copies of the field chain-of-custody record are made to allot one copy for each analytical procedure, plus one for moisture and one as a back-up.

5.1.3 Sample Logging

The field chain-of-custody record is used by the Sample Receipt Coordinator (SRC) to initiate sample logging procedures. Initial logging entries include field sample number, date of receipt at DCL, analyses requested, and comments on sample condition at the time of receipt as noted on the Sample Receipt Record. These are recorded in both a computer based log and in a bound logbook. After sample lotting is completed, the USATHAMA sample identification number for each sample and analysis is entered into the logs.

5.1.4 Sample Splitting

Following initial sample inspection, the SRC splits the samples into the required number of aliquots (one for each analytical procedure, one for moisture if the sample is a soil, and a large portion for back-up). The SRO properly labels the aliquots with the field sample identification number and the method of analysis, and relinquishes custody of the sample aliquots to the SRC.

5.1.5 Sample Lotting and Labeling

The number of samples which can be analyzed by a given method on a single day, as determined by the rate-limiting step in the analytical scheme, is designated as a "lot". The samples in a lot are labeled with a USATHAMA sample identification number consisting of a three letter lot code and individual three number sample designations (e.g. AAA001, AAA002). As split sample aliquots for a particular analytical procedure are received by the SRC, they are given the next alphabetical lot designation in sequence. Samples received and split at various times are grouped together in the same lot such that sample holding times are not jeopardized. The unique sample number is written in black permanent marker on white laboratory labeling tape, which is prominently placed on each sample container.

Quality control (QC) samples are a part of every lot, and are spiked according to the specific method requirements. The QC samples are provided upon request of the analyst.

5.1.6 Sample Storage

Samples are stored in a location appropriate to the holding requirements of the requested analytes. Heat-sensitive, light-sensitive, radioactive, or other samples having unusual physical characteristics or requiring special handling, are properly stored and maintained.

5.2 Chain-of-Custody

DCL maintains chain-of-custody records for all USATHAMA samples received at the laboratory.

A copy of applicable field chain-of-custody records is maintained with each sample lot. In addition, each lot of samples is maintained under a separate laboratory chain-of-custody record. The chain-of-custody includes unique sample number(s), date and time, source of sample(s), analyses required, signatures of relinquishing and receiving entities, and any other pertinent information. Copies of DCL's field and in-house chains-of-custodies for USATHAMA projects are provided in Appendix D.

5.3 Sample Handling Procedures

After samples have been received, split, and lotted, those not requiring extraction procedures are transferred to a central walk-in cold storage area. They are stored in this area until they are scheduled for analysis. Samples not requiring extraction procedures are prepared for analysis, within the required holding times, by the analyst or by a technician working under the direction of the analyst. These samples are usually analyzed within hours after preparation.

Samples which require extraction, distillation, or digestion procedures are prepared for analysis by the appropriate Inorganic or Organic Sample Preparation groups after lotting procedures have been completed. Extracts or distillates are stored in refrigerators in appropriate analytical areas of the laboratory.

The samples and extracts are maintained in their designated lots and under chain-of-custody, at all times. Separate preparation logbooks are maintained by the sample preparation groups to document sample handling.

5.4 Toxicity Characteristic Leaching Procedure

Samples which require Toxicity Characteristic Leaching Procedure (TCLP) are split and assigned a unique three-letter lot code. Chains-of-custody for these samples are signed off in the same manner as other samples requiring a USATHAMA-certified analysis. At the same time, chains-of-custody are printed (but not "initiated") for all prospective analyses to be generated from the TCLP leachate(s).

Once the original sample has been satisfactorily leached, both the chain-of-custody and any remaining original sample are transferred to Long Term Storage. The chains-of-custody for all generated leachates are now initiated by TCLP personnel. These leachates (along with their chains-of-custody) are stored and handled as any other USATHAMA samples which have been prepared for analysis.

The chains-of-custody for the original sample and the leachates are cross-referenced to facilitate traceability.

5.5 Holding Times

The holding times specified in DCL's USATHAMA Analyte Summary (Appendix B) are adhered to for all USATHAMA samples, extracts, distillates, and digestates.

5.6 Sample Analysis

5.6.1 Standards

Analytical standards are prepared either from Standard Analytical Reference Materials (SARMs) or Interim Reference Materials (IRM) supplied by USATHAMA, or from standard materials obtained by DCL from the EPA, the National Institute of Standards and Technology (NIST), or other commercial sources. Secondary standard materials may be used when SARM materials are available in only limited quantity. The secondary standards, which must be positively identified with an estimation of purity, are referenced to SARMs and periodically checked against them.

Standard materials procured from commercial sources other than USATHAMA, the Environmental Protection Agency (EPA), or the NIST are considered as "off-the-shelf" materials. The purity and identity of these materials is established from both analysis documentation supplied by the vendor and DCL analytical data. Materials are characterized by two independent methods whenever possible, including, but not limited to IR, GC, GC/MS, HPLC, and other inorganic techniques.

Metals are traceable to NIST, whenever possible. "Off-the-shelf" materials are characterized against EPA or NBS known standards whenever possible. All SARMS are stored in the quality control laboratory, under controlled access conditions. Generally, organic compounds are stored under refrigeration, while metals solutions are stored at room temperature.

5.6.2 Solutions

Analytical standard working solutions are normally prepared by the analyst performing the analysis, in accordance with the protocol defined in the approved analytical method. In some analytical procedures, a designated analyst prepares the standards, while other analysts carry out the procedure.

As new or replacement standard solutions are prepared, they are validated against either the previously used standard, a commercially prepared quantitative standard, or a standard prepared by another analyst for the purpose of validation.

Although validation acceptance criteria are established for each analytical method, protocol guidelines for acceptance of a new solution is that it is found, by analysis, to be within ±5% of the target value. All validations are documented either in the analyst's notebook or in a standards preparation logbook unique to USATHAMA and the analytical area using the standards.

5.6.3 Sample Preparation

Soil and water field samples are prepared for analysis according to the protocol defined in the analytical method for the specific analyte(s) being analyzed. Procedures for the preparation of mixed-matrix field samples, such as sediment, sludge, sewer, or lake-bottom samples, are discussed with USATHAMA on a case-by-case basis.

5.6.4 Instrument Calibration

The USATHAMA QA Program delineates, in detail, the requirements for instrument calibration for precertification, full method certification, initial calibration for analysis work, and daily calibration during sample analysis. DCL has implemented these guidelines for all USATHAMA work, as follows. Also see Section 4.3.6 (Certification) for additional details.

Instruments are tuned, as applicable, and the required number and concentrations of standards are analyzed daily with each lot of samples. Calibration criteria are either passed or corrective action is pursued by the analyst. If daily calibration criteria are not met, then initial calibration procedures are instituted to bring the analytical system back into calibration.

5.6.5 Initial Calibration

During initial calibration, a minimum of one blank and five calibration standards (Class 1) or one blank and three calibration standards (Class 1A and Class 1B) that bracket the certification testing range is analyzed singularly on one day. The concentrations of the calibration standards, in the solvent that results from all the preparation steps of the method, take into account any concentration steps that are part of the method. Concentrations in the solvent correspond to those in an environmental matrix as if the method preparation steps had been performed.

In addition to the initial calibration standards, Class 1 and 1B methods require the analysis of calibration check standards (Section 5.6.7). During a Class 1 or Class 1B initial calibration, a calibration check standard is analyzed at the completion of calibration. If the method requires what could be an initial calibration each day analysis is performed, then the calibration check standards are analyzed once a week rather than each day.

If the results of the calibration check standard are not acceptable, immediate reanalysis of the calibration check standard is required. If the results of the reanalysis still exceed the limits of acceptability, the system is considered to have failed calibration. Sample analysis is halted and will not resume until successful completion of initial calibration. Corrective actions taken to restore initial calibration are documented in the analysts' notebook.

5.6.6 Daily Calibration

Calibration standards are analyzed each day to verify that instrument response has not changed from previous calibration. Each day before sample analysis, the highest concentration standard is analyzed. The response must fall within the required percentage or two standard deviations of the mean response for the same concentration, as determined from precertification, certification, and prior initial/daily calibrations. If the response fails this test, the daily standard is reanalyzed. If the response from the second analysis fails this range, initial calibration is performed before analyzing samples.

Each day after sample analyses are completed, the highest concentration standard is analyzed. If the response is not within the required percentage or two standard deviations of the mean response from precertification, certification, and prior initial/daily calibrations, the daily standard shall be reanalyzed. If the response from the second analysis fails the range, the system is considered to have failed calibration. Initial calibration is performed and all samples analyzed since the last acceptable calibration are reanalyzed.

For non-linear or non-zero-intercept calibration curves, daily calibration consists of analysis of the low, middle, and high standards at the beginning of the day. When sample analyses are completed at the end of the day, the low and high standards are analyzed. Instrument responses for each concentration determination must fall within two standard deviations of the mean response, as described previously, for the appropriate standard. For calibrations fitted by the quadratic equation, a minimum of four standards over the certified range are required and the highest level standard analyzed at the end of the day. For all other equations, one more standard than needed to meet the degrees of freedom for any lack-of-fit is required, as a minimum.

5.6.7 Calibration Check Standards

Calibration check standards are required for all Class 1 and 1B methods and are analyzed during precertification and with each initial certification. The calibration check standard contains all analytes of interest for the method in question at a concentration near the upper end of the calibration range. Results of the calibration check standards shall fall within the limits of acceptability as described below:

CASE 1.
A certified check standard is available from the EPA or some other source with both the true value and limits of acceptability specified by the supplier. The results must fall within the limits specified by the supplier, or +/-10 percent for inorganics, +/-25 percent for organics, whichever is less.

CASE 2.
A certified check standard is available from the EPA or some other source with a true value specified but without limits of acceptability. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

CASE 3.
If no certified check standard is available, the contractor laboratory shall prepare a check standard using a second source of reference material. This standard shall be prepared by a different analyst than the one who prepared the calibration standard. If weighing of the material is required, a different balance should be used, if possible. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

CASE 4.
If there is only one source of reference material available, then the calibration and calibration check standards must be prepared from the same material. The standards shall be prepared by different analysts. If weighing is required, different balances should be used, if possible. The results must fall within +/-10 percent for inorganics and within +/-25 percent for organics.

For all cases listed above, after the seventh acceptable calibration check standard, the limits of acceptability are +/- two standard deviations, as determined from the first seven points.

For multi-analyte methods, the calibration check standard contains all analytes of interest. For the check standard to be deemed acceptable at least 2/3 of the analytes must meet the limits of acceptability as defined above (also see Table 3). In addition, if a single analyte falls outside the limits of acceptability for two consecutive times, then the calibration check standard is deemed unacceptable. If a calibration check standard is not acceptable, the procedures detailed above are followed.

Table 3.
MINIMUM NUMBER OF IN-CONTROL POINTS FOR MULTI-ANALYTE METHODS

Required Control Analytes Per Method	Required Number of Data Values Falling Between the UCL and LCL
1	1
2	2
3	2
4	3
5	4
6	4
7	5
8	6
9	6
10	7
11	8
12	8
13	9
14	10
15	10
16	11
17	12
18	12
19	13
20	14
21	14
22	15
23	16
24	16
25	17

5.6.8 Analytical Procedures

All field samples are analyzed according to approved, laboratory certified USATHAMA analytical methods. All deviations shall be approved by USATHAMA prior to implementation. These deviations are also documented in the analyst's notebook.

5.6.9 Second-Column Confirmation

In several GC and HPLC methods (e.g., organochlorine pesticides and explosives), the presence of compounds is routinely confirmed on a second column. The confirmation is usually performed on the basis of a Class 2 certification. Confirmation does not necessarily have to be performed within holding times, but must be accomplished within ten (10) days of sample analysis.

5.7 Data Handling

Although the primary emphasis of the USATHAMA QA Program is the control of sample analysis and the handling of data, record keeping maintains its importance in the overall assessment of the production of quality of data and is used in part to document the control of sample analysis. The degree of rigor used in documenting sampling and analysis activities cannot be understated. All activities require extensive documentation and special handling protocols. All activities are to be performed under chain-of-custody procedures. Particularly in these situations, the attitude is: "If you didn't write it down, you didn't do it."

For most USATHAMA projects, this degree of documentation is required. For some projects, documentation in the form of an EPA CLP package is required. In any case, the records described in this Quality Assurance document shall be maintained and will be available for inspection by USATHAMA.

5.7.1 Data Reduction

Generally, data have been collected during the analysis of samples either into computer based data files or onto hard copy sheets, which, in turn, are either machine generated or hand written. All of the data are eventually compiled in computer files. The data pertaining to analytical standards are either compared to the most recent initial calibration curve, in the case of a daily calibration, or used to generate new initial calibration curves, in accordance with those generated during pre-certification. The appropriate standard curve is used to evaluate the field sample data to determine the amount of analyte present. Finally, all of the computer generated calculations are generated as hard copy output.

5.7.2 Data Validation

Initial data validation is accomplished during data collection through the use of quality control samples and calibration check standards. Errors detected through a review of these monitors by Quality Assurance during analysis are corrected during the data collection phase of the analysis. Only analytically valid data are processed further.

Following an analyst's computer-based reduction of data and production of a numerical results report, the entire assemblage of data is given to a peer analyst for review and validation. The peer analyst checks that the analytical method was followed, that there are no errors in the transcription of data, that the best-fit curve was used, and that the numerical report of data contains no calculation or transcription errors.

The data package is then reviewed by the appropriate Group Leader or Section Manager. The data report is particularly scrutinized to assure that all reported data values are in the proper range or have dilution factors, that the method has been carefully followed, that instrumentation was properly tuned or calibrated, and that the instrumental data was properly interpreted. A general review of the data package is also made to assure that all required documentation is present.

The final step in data validation is the review by Quality Assurance. The content of each data package is closely checked for errors or omissions that would negatively impact on the admissibility of the data in litigation proceedings. Corrective action is initiated and documented as outlined in section 10.0.

5.7.3 Data Reporting

The results for samples analyzed for USATHAMA projects are entered into the USATHAMA-provided software program (IRDMS). Data created using the IRDMS can then be electronically transmitted to USATHAMA Via Potomac Research Inc. (PRI), or a diskette together with hard copy printouts can be submitted.

Data is entered on a coding form by the analyst, which is verified by the peer checker and, group leader/section manager. QA personnel review data for obvious errors. These data are encoded onto a diskette, checked through two USATHAMA software routines, then printed out and verified by visual inspection by a Data Entry Specialist. Verified analytical results are then submitted to USATHAMA. DCL retains a copy diskette of all data submitted.

All information pertaining to the analysis of a lot of samples is collected into a data package at the completion of analysis. The contents of data packages varies with methods of analysis. The package is reviewed by Quality Assurance to eliminate technical errors that might affect the litigation quality of the data. The reported data is also reviewed by Data Entry for completeness before release.

All data packages are archived at DCL until a task or delivery order at a particular installation is complete. At that time, all pertinent documentation filed in appropriately-labeled boxes is delivered either to USATHAMA directly, or to the prime contractor responsible for final review of the data packages. In the second case, the prime contractor is responsible for the delivery of DCL data boxes to USATHAMA.

6.0
ANALYTICAL SYSTEM CONTROLS

6.1 Sample Control

As discussed in the section of this QA Plan on Sample Management, DCL is not generally responsible for the collection of samples from sites in the field. However, DCL efforts in sample control may extend into field sample collection. As directed by USATHAMA or the prime contractor, DCL provides proper sample collection bottles, sample preservatives, labeling material, sample shipping containers (coolers), and technical assistance to field sample collection crews. DCL also works in concert with USATHAMA or the prime contractor on sample shipping and receiving.

Samples received at DCL are under the control of Sample Receipt personnel from receipt at the lab to acceptance by an analyst for extraction or preparation. Samples are not released for processing until all documentation is completed and the samples are properly lotted and labeled. Holding times are closely monitored by the analysts, Sample Receipt and laboratory management.

DCL Project Managers communicate regularly with USATHAMA and/or other involved prime contractors to alleviate sample shipping, holding time, and analysis difficulties.

6.2 Document Control

Document control is primarily the responsibility of Quality Assurance. Sample documents generated in the field during sample collection and shipping are maintained in QA files. Laboratory chain-of-custody records, sample receipt and tracking records, data reporting forms and analysis data packages, and corrective action records are maintained by Quality Assurance. On a schedule determined by contract requirements, QA also archives or otherwise controls all bound notebooks and logbooks containing data pertinent to USATHAMA work.

6.3 Quality Control Samples

Quality control chemists within the Quality Assurance Section of DCL prepare most of the quality control samples required during sample analysis. These samples are prepared from USATHAMA-supplied SARM and IRM stocks, and other reference materials. Other reference materials include EPA, and NIST standard materials, and "off-the-shelf" materials. "Off-the-shelf" materials are analyzed by DCL, with positive identification and estimate of purity, with EPA standard reference materials, where possible, using at least two different methods.

Quality control stock and dilute working solutions are prepared and maintained separately from those used by analysts as standards. Exceptions to this procedure are made only when primary stock material is in very short supply, or when the primary solution is unstable. In these cases, the same primary solution is used to prepare separate dilute working solutions. Samples are prepared in accordance with parameters defined in each analytical method. These parameters include the control analytes, the concentration levels at which the analytes should be spiked, control sample matrix, spike equilibration time, and procedures for preparation of the sample for analysis.

Quality control samples which are not regularly prepared by the quality control chemists include surrogate spiking solutions and spiked samples required in the GC/MS methods for volatile and semi-volatile organic compounds. These surrogate preparations are handled by the GC/MS Group and the Extraction Group, respectively.

Quality control samples are included in every lot of USATHAMA samples, as required in the USATHAMA QA Program and specified in each certified analytical method. The control samples are processed through the entire analytical method and quantitated on the same calibration curve as the field samples. The results for the quality control samples are evaluated first by the analyst, and then by Quality Assurance, to determine their acceptability.

Calibration check standards are prepared by someone other than the person preparing the standards. Calibration check standards are analyzed at the time of an initial calibration, or once per week when routine initial calibrations replace daily calibrations. The analysis results must meet the criteria established by their originator.

6.4 Control Charts

For Class 1, Class 1A, and Class 1B certified methods, control charts are used to monitor the variations in the precision and accuracy of routine analyses and to detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts is not adjusted for accuracy. Control charts are not used with Class 2 certified methods.

Control charts include the analyte, method number, DCL laboratory code of UB, spike concentration, and chart title. All data presented on a control chart are also presented in tabular form. The following charts may be selected from the USATHAMA-supplied computer control chart program:

1. Single-Day X-Bar Control Chart (High Spike Conc.)
2. Single-Day Range Control Chart (High Spike Conc.)
3. Three-Day X-Bar Control Chart (Low Spike Conc.)
4. Three-Day Range Control Chart (Low Spike Conc.)

In addition, the following information is also included on each control chart:

- Three-letter lot designation for each point, shown on the x-axis;
- Percent recovery (for X-bar control charts), or range (for R control charts) along the y-axis;
- Upper control limit (UCL);
- Upper warning limit (UWL);
- Mean;
- Lower warning limit (LWL), on X-bar charts; and
- Lower control limit (LCL), on X-bar charts.

For some analytes specified by USATHAMA, warning limits on X-bar charts are deleted and replaced by modified control limits based upon data quality specifications.

6.4.1 Control Chart Plotting: Single-Day

The initial control chart is prepared using the four days of certification data closest to the spiking concentration used during analysis. The average (X-bar), average range (R), and control limits for both are updated after each in-control lot for the first 20 lots. Limits established after lot 20 are used for the next 20 lots. Control charts are updated after each 20 lots thereafter, using the most recent 40 points. In interpreting the control charts developed for the initial lots (1-20), the limits established from the previous lots are used to control the current lot.

When modified limits are established, data for samples are accepted if the control data fall between the modified limits. If modified limits have not been established, data for samples are accepted, based upon the recoveries established during certification and the current performance of the method. In updating the control charts, the new data must be combined with the individual values of previous average percent recoveries and not the mean of all previous data. Only lots evaluated as in-control are applicable to the 20 and 40 lot requirements for establishing and updating control chart limits. Out-of-control or outlier points are plotted; however, such lots are not utilized in lot number requirements or control chart calculations.

All recoveries are plotted, whether or not the lot is in-control. Plotted points represent averaged instrument measurements and not the individual measurement values. Each individual recovery measurement value is tested as an outlier using Dixon's Test at the 98% confidence level. If the datum is not classified as an outlier by the test, the point is included in updating the control chart limits. If the datum is classified as an outlier, it is not used in updating the control chart limits. Range data are not subject to outlier testing.

After the first 20 in-control sample lots, control limits are recalculated using only in-control data points. The control limits are then drawn backward to encompass all previous points. Any points falling outside the control limits (UCL or LCL) are dropped from the calculations (but left on the charts) and the control limits recalculated using only points between those limits. This practice of dropping points and recalculating limits is performed only once, at the initialization of stable limits. Charts are then updated with newly calculated control limits and all points plotted.

6.4.2 Three-Point Moving Average

Analytical data for analytes prepared in the single low concentration QC sample are plotted and evaluated on a three-day-moving-average control chart. Data for the surrogates spiked in a standard matrix and used in GC/MS analyses are also charted on a three-day-moving-average control chart. Plotting criteria for the three-point moving average control charts are similar to those described above (Section 6.4.1) for single-day control charts. Data for analytes prepared in duplicate QC samples at high concentrations are plotted and evaluated on single-day control charts.

Computer generated control charts maintained by Quality Assurance are updated and printed weekly, while analysts plot data points by hand as sample lots are analyzed. This allows for both computer maintenance and evaluation of a large data base with software calculation of control limits, and immediate daily surveillance of analytical trends.

6.5 Out-of-Control Conditions

Results of the analysis of quality control samples are reported to QA within 48 hours of completion through the analyst's submission of a Preliminary QC Report.

The analyst quantifies each analyte in the method blank and spiked QC sample each day of analysis. Processing of additional lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control.

An indication of an out-of-control situation may include: A value outside the control limits or classified as outlier by statistical test; A series of seven successive points on the same side of the mean; A series of five successive points going in the same direction; A cyclical pattern of control values, or; Two consecutive points between the UWL and UCL or the LWL and LCL.

If the points for at least two-thirds of the control analytes for a multi-analyte method are classified as in-control, the method is in control and environmental sample data may be reported. A method may be deemed out-of-control even if greater than or equal to 2/3 of the control analytes meet control criteria. Of the remaining control analytes (less than 1/3 possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is deemed out-of-control. If data points for fewer than 2/3 of the control analytes are classified as in control, the method is considered to be out-of-control and all work on that method must cease immediately. No data for environmental samples in that lot may be reported.

In all cases, investigation by the analyst and the Quality Assurance Coordinator is required to determine the cause of the condition and to decide on appropriate corrective action. The pertinent details of the situation and the corrective action taken are fully documented in a Corrective Action Report (CAR). (See also section 10.0.) Field sample data effected by the situation are evaluated and reanalyzed as necessary.

When a method is determined to be out of control, the analysis of field samples by that method is suspended. Corrective action must be documented and the method must be demonstrated to be in control before analysis of field samples is reinstated. Analytical control is demonstrated through the acceptable analysis of an appropriate set of QA samples.

7.0
PREVENTATIVE MAINTENANCE

All analytical instrumentation used at DCL is maintained to provide consistent, high-quality performance. Most instruments are maintained by the manufacturer, under contract. Each instrument is labeled with a unique number and instrument information peculiar to USATHAMA requirements. Instrument service records and maintenance calibrations are maintained by the appropriate section and in a logbook unique for each instrument.

The primary objective of the instrument maintenance program is to assure the quality of the analytical data generated by the instrument. While there are analytical systems which require absolute calibration, such as balances, the majority of analytical systems used by DCL for the analysis of USATHAMA samples are calibrated at the time of use by the analyst. This is accomplished through generation of a chemical calibration curve, based upon instrument response verses analyte concentration. This curve is used to evaluate field sample data through instrument responses.

Major instrument systems which are calibrated on an "as used" basis are maintained under either an "on call" or a preventative maintenance contract with the manufacturer. Preventative maintenance is scheduled in each instrument contract. When an instrument cannot perform to specifications and DCL technicians cannot return it to specification, a contracted repair service (usually the manufacturer) is called.

Instrument systems which must maintain an absolute calibration, such as analytical balances, are serviced under contract with the manufacturer, usually on an annual basis. Balances are also checked, on at least a weekly basis, for accuracy by Quality Assurance, using NIST-traceable weights. Temperatures of freezers, refrigerators, and walk-in coolers are recorded every working day by QA. When temperatures are noted outside the acceptable range, appropriate personnel are notified for correction. Ovens are calibrated and their temperatures maintained regularly by the appropriate section personnel.

8.0
RECORDKEEPING

8.1 Laboratory Notebooks

Bound, sequentially-numbered laboratory notebooks with pre-numbered pages are utilized by all analysts for analytical recordkeeping. Notebooks are generally issued to and used by an individual analyst. Any loose sheets of data which must be included in a notebook are securely taped into the notebook and signed and dated across the edges, halfway on the inserted sheet and halfway on the notebook page. Each data page is signed and dated by the analyst entering data on that page, as well as reviewed, signed, and dated by a witness. All entries are required to be in black ink. Corrections are made by a single strikeout, which is dated and initialed.

8.2 Logbooks

8.2.1 General

Individual logbook entries are signed and dated by the analyst or technician making the entry. These notebooks include, for example, instrument use and maintenance/calibration logs, pH logs, sample moisture determination logs, and sample receipt logs.

Recordkeeping for sample receipt is discussed under the Sample Management Section 5.1.

8.2.2 Standards

A bound logbook is maintained for all analytical reference materials used for USATHAMA work. The record includes the date of receipt, preparer, source, purity, composition, storage requirements, and expiration date, if applicable. Characterization data for purchased reference material is also included.

The preparation of working standards from reference materials is recorded in a bound logbook. This logbook may be of general use by several analysts for USATHAMA standards preparation, or an individual analyst's notebook, as for preparation of standards used for a single analytical run associated with a single lot of samples.

8.2.3 Instrument

Instrument maintenance records and, where applicable, instrument tuning and calibration data, are maintained in instrument specific logbooks. Actual analytical conditions pertaining to an individual lot analysis are recorded in the analyst's notebook, along with other pertinent analytical information.

8.3 Hard-Copy Output

Hard-copy output, (e.g., chromatograms and computer generated data evaluations) is labeled with date, time (where applicable), analytical method, sample numbers, the name or initials of the analyst generating the output, and other pertinent information. Storage of hard-copy output is with related analytical data pertaining to an individual lot analysis. All such data, comprising a complete record of an analysis, are compiled into one or more envelopes for archiving. The envelopes are properly labeled with the lot designation, method of analysis, matrix, analyst, analyst's notebook, and date of completion. When samples from multiple sites or projects are grouped together in a single lot, the data pertaining to each site are compiled (or copied) and stored separately, as directed by USATHAMA. All copies indicate the location of the original data.

9.0
AUDITS

DCL facilities are always available for any required audits, announced or unannounced, by USATHAMA representatives.

The DCL Quality Assurance Coordinator conducts internal audits of critical functions within the laboratory, including verification that record keeping procedures are adequate, verification that general good laboratory practices, analytical methods and standard operating procedures are being followed, and continual assessment of quality control sample results. A summary of such audits is available for review at the laboratory.

10.0
CORRECTIVE ACTION

When, as a result of audit procedures or the analysis of quality control samples, the analytical or other laboratory systems are found to be unsatisfactory, a corrective action is initiated. The unsatisfactory situation may be either immediate or long term in nature. Immediate short term problems may include unsatisfactory performance on quality control samples (which may be more involved than simply out-of-control data), errors or omissions in the compilation of the data package, or other problems peculiar to a single lot of samples. Long-term problems include trends or cycles in quality control sample analysis data, standard and solution preparation control, staff training in analytical and quality control procedures, or other problems which affect several analytical methods or multiple lots of samples.

To enhance the timeliness of corrective action and thereby reduce the generation of unacceptable data, problems identified by assessment procedures are resolved at the lowest possible management level. Problems that cannot be resolved at this level are reported to the Quality Assurance Coordinator (QAC) for resolution. The QAC determines the management level at which the problem can best be resolved, and notifies the appropriate manager. Weekly progress reports detail all problems and subsequent resolutions.

Steps included in the corrective action system include:

1. Defining the problem;
2. Assigning responsibility for problem investigation;
3. Investigating and determining the cause of the problem;
4. Assigning responsibility for problem resolution; and
5. Verifying that the resolution has corrected the problem.

Problems requiring corrective action may not be easy to identify or define. The situation may not be producing out-of-control data, but simply producing data not of the quality desired. The project manager, section managers, analysts, and the quality assurance staff combine efforts in solving long-term unsatisfactory situations.

All corrective actions are documented by Quality Assurance. Final corrective action reports, which relate to a particular lot analysis, are included in the data package for that lot.

11.0
QUALITY CONTROL REPORTS

DCL provides weekly quality assurance evaluation reports to USATHAMA, in conjunction with weekly interim technical reports from project management. The QA reports include charts and tables of quality control data, a control chart checklist delineating contracts and lots, and copies of Corrective Action Reports (CARs). These CARs include explanations of analytical or quality control problems and discussions of the corrective actions taken to alleviate those problems. Observations of data trends or situations which could develop into problems are also discussed in this report, as well as preliminary acceptance or rejection of analytical data.

APPENDIX A

APPENDIX A

LACK OF FIT AND ZERO INTERCEPT TESTS

LACK OF FIT TEST FOR CALIBRATION CURVES AND CERTIFICATION DATA

For most routinely used analytical systems, the instrument response is assumed to be a linear function of analyte concentration. The linear model can be tested by analyzing standards that have been prepared in replicate at each concentration. In addition to the calibration data (target versus instrument response), certification data (target versus found) is also subjected to the Lack of Fit (LOF) test. The usual method of least squares fitting assumes no error in the concentrations of standards.

There are two distinct linear first-order regression models that are generally encountered in analytical calibration. The non-zero intercept model is the most familiar, given by:

$$Y = Y_o + bX$$

where:

Y = Dependent Variable (Instrument Response or Found Concentration);

Y_o = Y Axis Intercept;

b = Slope of the Line; and

X = Target Concentration.

The estimates Y_o and b are calculated to minimize the Sum of Squares (SS) of the deviations from the line without restrictions. For some analyses, however, theory predicts that the response of the instrument should be linear with concentration and should also be zero when there is no analyte present. Thus, if the instrument has been calibrated correctly, the calculated line should pass through the origin by definition. The proper regression model would then be the Zero Intercept model:

$$\hat{Y} = b_o X$$

where:

$\hat{Y}$ = Predicted Value of Dependent Variable;

b_o = Slope of Line Through Origin; and

X = Target Concentration.

The estimate of b_o is calculated to minimize the SS of deviations from the line with the restriction that the line must pass through the origin.

For the model with an intercept:

$$b = \frac{N\sum X_i Y_i - \sum X_i \sum Y_i}{N\sum X_i^2 - (\sum X_i)^2} \qquad Y_o = \frac{\sum Y_i - b\sum X_i}{N}$$

For the model through the origin:

$$b_o = \frac{\sum X_i Y_i}{\sum X_i^2} \qquad Y_o = 0$$

where:

N = Number of Data Points;

X_i = i-th Target Concentration; and

Y_i = i-th Value of Dependent Variable.

The correlation coefficient is a measure of the relationship between two independent variables. In calibration and certification problems, it is assumed that a definite functional relationship exists between the dependent (response or found concentration) and independent (target concentration) variables. Therefore, the correlation coefficient is an insensitive tool for evaluating the quality of the fitted equation.

A more sensitive tool for evaluating the fitted equation is a regression analysis, in which the sources of variation are fractionated into the SS attributable to regression and the SS for residuals. When replicate measurements have been made, the residual SS can be separated into a systematic error component and a random error component. The SS due to systematic error is designated the SS due to LOF because it arises from the inadequacy of the fitted regression model to describe the experimental points.

For the model with intercept, the equation for calculating the SS of residuals is:

$$\text{SS Residual} = \left[\sum Y^2 - \frac{(\sum Y)^2}{N} \right] - b^2 \left[\sum X^2 - \frac{(\sum X)^2}{N} \right]$$

where:

Y = Values of Dependent Variable;

X = Target Concentration;

N = Total Number of Measurements; and

b = Slope of Best Fit Line.

The number of degrees of freedom (df) is N - 2, because two regression coefficients were fitted (slope and Y-axis intercept).

The SS for random error is independent of the regression model employed, depending only on the distribution of replicates around the mean at each concentration. When duplicate measurements have been acquired at each concentration, the SS for random error is given by:

$$\text{SS Random Error} = \frac{\sum d^2}{2}$$

where:

d = Difference in Values for Each Set of Duplicates.

The total df in this error estimate would be equal to the number of duplicates sets because each would contribute 1 df (2 - 1 = 1). When more than two replicates measurements are made, the SS random error for each set is given by:

$$\text{SS Random Error} = \sum Y^2 - \frac{(\sum Y)^2}{n}$$

where:

n = Number of Replicates in Each Set (df is n - 1).

Both the SS random error and the df are then summed across all sets to get the total SS random error and the total df.

After the total SS random error has been calculated, the SS for LOF can be obtained by difference according to:

$$\text{SS LOF} = (\text{SS Residual}) - (\text{Total SS Random Error})$$

Similarly, the df associated with LOF is given by:

$$\text{df LOF} = (\text{df Residual}) - (\text{df Total Random Error})$$

Regression analysis tables are used to determine whether the data fit the linear models and which linear model is more appropriate. The tables are calculated as shown in Table A-1. For calibration curves and certification data, the replicate analyses of the blank (zero concentration) are not used to obtain regression equations.

After calculating the regression analysis table, the F-ratio for LOF is compared to an F Table (Table A-2) to determine if the regression model is an adequate description of the data. The df LOF is used as v_1, df random error for v_2, and 95 percent confidence level. If the calculated F-ratio exceeds the value in the table, there is statistically significant LOF and the data are not linear.

The nature of this test is such that large random error will mask nonlinearity in the data. Very small random error can cause very small (and possibly unimportant) nonlinearity to be found significant (e.g., significant LOF). In fact, when random error is large (or very small), it is difficult to detect systematic variations that might cause LOF.

Table A.1. Regression Analysis Table for Model with Intercept

Source of Variation	Sum of Square (SS)	Degrees of Freedom (df)	Mean Square (MS)	F-Ratio
Residual	$\left[\Sigma Y^2 - \frac{(\Sigma Y)^2}{N}\right] - b^2\left[\Sigma X^2 - \frac{(\Sigma X)^2}{N}\right]$	N-2	$\frac{\text{Residual SS}}{N-2}$	-
Individual Error (for each set of data at each concentration)	$\Sigma Y^2 - \frac{(\Sigma Y)^2}{n}$ (for duplicates -- $\frac{\Sigma d^2}{2}$)	n-1	-	-
Total Error	Σ Individual Error SS	Σ df for Individual Error	$\frac{\text{Total Error SS}}{\text{df Total Error}}$	-
Lack of Fit (LOF)	Residual SS - Total Error SS	df Residual - df Total Error	$\frac{\text{LOF SS}}{\text{df LOF}}$	$\frac{\text{MS LOF}}{\text{MS Total Error}}$

where Y = Values for Dependent Variable
X = Target Concentration
N = Total Number of Measurement
n = Number of Replicates at each Concentration
d = Difference between Duplicates

Do not round off intermediate numbers in calculations. Carry through at least six digits to avoid rounding off errors, even though in the final results less than six digits will be significant.

Table A.2. F-Ratio Critical Values (From Scheffe, 1959)

THE ANALYSIS OF VARIANCE

UPPER α POINT* OF F WITH ν_1 AND ν_2 D.F.

$\alpha = 0.05$

ν_2 \ ν_1	1	2	3	4	5	6	7	8	9
1	161	200	216	225	230	234	237	239	241
2	18.5	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4
3	10.1	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02
11	4.84	3.98	3.59	3.36	3.20	3.09	3.01	2.95	2.90
12	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.80
13	4.67	3.81	3.41	3.18	3.03	2.92	2.83	2.77	2.71
14	4.60	3.74	3.34	3.11	2.96	2.85	2.76	2.70	2.65
15	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59
16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54
17	4.45	3.59	3.20	2.96	2.81	2.70	2.61	2.55	2.49
18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.46
19	4.38	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.42
20	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39
21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.37
22	4.30	3.44	3.05	2.82	2.66	2.55	2.46	2.40	2.34
23	4.28	3.42	3.03	2.80	2.64	2.53	2.44	2.37	2.32
24	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.30
25	4.24	3.39	2.99	2.76	2.60	2.49	2.40	2.34	2.28
26	4.23	3.37	2.98	2.74	2.59	2.47	2.39	2.32	2.27
27	4.21	3.35	2.96	2.73	2.57	2.46	2.37	2.31	2.25
28	4.20	3.34	2.95	2.71	2.56	2.45	2.36	2.29	2.24
29	4.18	3.33	2.93	2.70	2.55	2.43	2.35	2.28	2.22
30	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21
40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.12
60	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	2.04
120	3.92	3.07	2.68	2.45	2.29	2.17	2.09	2.02	1.96
∞	3.84	3.00	2.60	2.37	2.21	2.10	2.01	1.94	1.88

* Rounded off to three significant figures from tables of M. Merrington and C. M. Thompson in *Biometrika*, Vol. 33, pp. 78–87, 1943. Reproduced with the kind permission of the authors and the editor.

A.2 ZERO INTERCEPT TEST FOR CALIBRATION CURVES AND CERTIFICATION DATA

If the linear model with intercept is acceptable, the intercept must be tested to determine if it is significantly different from zero. The expression for calculating the slope of the line through the origin is:

$$b_o = \frac{\sum X_i Y_i}{\sum X_i^2}$$

Before testing the hypothesis that the intercept is zero, a regression analysis table is constructed (Table A-3). If the LOF for the model through the origin is not statistically significant, the Zero Intercept hypothesis is tested using the differences between the residual SS for the intercept and origin models.

To test the hypothesis that the intercept does not differ significantly from zero, calculate:

$$F = \frac{\text{SS Residual for Zero Intercept Model} - \text{SS Residual of Model with Intercept}}{\text{MS Residual of Model with Intercept}}$$

The df in the numerator will always be 1 because (N - 1) - (N - 2) = 1 and, therefore, the difference in these SS are divided by 1 to get the MS. The df in the denominator is N - 2.

The calculated F-ratio is compared to the critical values of F in Table A-2, at $v_1 = 1$ and $v_2 = N - 2$. If the calculated F-ratio is less than the critical value, the Zero Intercept model is accepted.

Generally, certification data will be expected to have intercepts not statistically different from zero. The procedures for daily calibration assume that the zero intercept model can be accepted. If intercepts are statistically different from zero, more rigorous calibration controls will be required and will be specified on a case-by-case basis in the project QC plan.

Table A.3. Regression Analysis Table for Model Through the Origin

Source of Variation	Sum of Square (SS)	Degrees of Freedom (df)	Mean Square (MS)	F-Ratio
Residual	$\Sigma Y^2 - \frac{(\Sigma XY)^2}{\Sigma X^2}$	N-1	$\frac{\text{Residual SS}}{\text{N-1}}$	-
Individual Error (for each set of data at each concentration)	$\Sigma Y^2 - \frac{(\Sigma Y)^2}{n}$ (for duplicates -- $\frac{\Sigma d^2}{2}$)	n-1	-	-
Total Error	Σ Individual Error SS	Σ df for Individual Error	$\frac{\text{Total Error SS}}{\text{df Total Error}}$	-
Lack of Fit (LOF)	Residual SS - Total Error SS	df Residual - df Total Error	$\frac{\text{LOF SS}}{\text{df LOF}}$	$\frac{\text{MS LOF}}{\text{MS Total Error}}$

where Y = Values for Dependent Variable
X = Target Concentration
N = Total Number of Measurement
n = Number of Replicates at each Concentration
d = Difference between Duplicates

Do not round off intermediate numbers in calculations. Carry through at least six digits to avoid rounding off errors, even though in the final results less than six digits will be significant.

APPENDIX C

APPENDIX C

SAMPLE CONTAINER CLEANING PROCEDURES

To ensure the integrity of aqueous and solid samples, steps must be taken to minimize contamination from the containers in which they are stored. If the analyte(s) to be determined are organic in nature, the container should be made of amber glass. If the analyte(s) are inorganic, the container should be polyethylene. When both organic and inorganic substances are expected to be present, separate samples should be taken. New sample bottles must be cleaned according to either of the procedures presented below; reuse of sample containers is expressly prohibited. The procedure that was used must be documented. Commercially cleaned containers may be utilized if cleaning procedures comply with those provided in this appendix and prior USATHAMA Chemistry Branch approval is obtained. The procedures for cleaning the glass and polyethylene containers and their caps are as follows:

ALTERNATE A:

- Polyethylene Bottles and Polyethylene Caps

  (1) Rinse bottles and lids with 5 percent sodium hydroxide.

  (2) Rinse with deionized water.

  (3) Rinse with 5 percent Ultrex (or equivalent) nitric acid in deionized water.

  (4) Rinse with deionized water.

  (5) Drain and air dry.

- Amber-Glass Bottles or 40-ml Vials

  (1) Scrub and wash bottles in detergent.

  (2) Rinse with copious amounts of distilled water.

  (3) Rinse with acetone.

  (4) Rinse with methylene chloride (Nanograde or equivalent).

  (5) Rinse with hexane (Nanograde or equivalent).

  (6) Air dry.

(7) Heat to 200°C.

(8) Allow to cool.

(9) Cap with clean caps with Teflon liners.

- Bottle Caps

(1) Remove paper liners from caps.

(2) Wash with detergent.

(3) Rinse with distilled water.

(4) Dry at 40°C.

- Teflon Liners (avoid contact with fingers)

(1) Wash with detergent.

(2) Rinse with distilled water.

(3) Rinse with acetone.

(4) Rinse with hexane (Nanograde or equivalent).

(5) Air dry.

(6) Place liners in cleaned caps.

(7) Heat to 40°C for 2 hours.

(8) Allow to cool.

(9) Use to cap cleaned bottles.

ALTERNATE B: (Specified by EPA for CLP)

- Amber Glass Bottles

(1) Wash containers, closures, and teflon liners in hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse with 1:1 nitric acid.

(4) Rinse three times with ASTM Type 1 deionized water.

(5) Rinse with pesticide grade methylene chloride.

(6) Oven dry.

(7) Remove containers, closures, and teflon liners from oven.

(8) Place teflon liners in closures and place closures on containers. Attendant to wear gloves and containers not to be removed from preparation room until sealed.

- 40 mL Borosilicate Glass Vials

(1) Wash vials, septa, and closures in hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse three times with ASTM Type 1 deionized water.

(4) Oven dry vials, septa, and closures.

(5) Remove vials, septa, and closures from oven.

(6) Place septa in closures, teflon side down, and place on vials. Attendant to wear gloves and vials not to be removed from preparation room until sealed.

- High Density Polyethylene Bottles

(1) Wash bottles, closures, and teflon liners with hot tap water with laboratory grade non-phosphate detergent.

(2) Rinse three times with tap water.

(3) Rinse with 1:1 nitric acid.

(4) Rinse three times with ASTM Type 1 deionized water.

(5) Air dry in contaminant-free environment.

(6) Place liners in closures and place closures on bottles. Attendant to wear gloves and bottles not to be removed from preparation room until sealed.

Documentation must be provided to the USATHAMA Chemistry Branch validating that the bottles are in fact "clean." Documentation may consist of the results of "bottle blank" analysis using the method(s) that will be applied to the sample that will be placed in that bottle. QC results from the supplier of commercially cleaned containers, demonstrating that the bottle(s) are "clean," will be acceptable. The documentation must be provided before the bottles are used to collect samples in the field. This validation is to be performed or provided for each batch or "lot" of bottles cleaned together and must be provided at least once for each installation where they are used.

APPENDIX D

DATA CHEM

PAGE _____ OF _____

PRIME CONTRACTOR	PRIME CONT. CODE	BASE CLOSURE YES NO
INSTALLATION	INSTAL. CODE	MATRIX

FIELD CHAIN-OF-CUSTODY

for Samples Collected under
USATHAMA QA/QC

FILE TYPE	SITE TYPE	SITE IDENTIFICATION	FIELD SAMPLE NO.	SAMPLE DATE	SAMPLE PROGRAM	SAMPLE DEPTH	TQ.	NO. OF CONTAINERS	PARAMETERS/METHOD NUMBERS FOR ANALYSIS												

Remarks/Comments:	Airbill No.	Shipped by: Fed Ex _____ UPS _____ Other _____

Sampled by:	Date/Time:	Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:	Relinquished by:	Date/Time:

Data Chem Laboratories 960 West LeVoy Drive/Salt Lake City, Utah 84123

THIS LOT DIES
Extraction: _____
Analysis: _____

CHAIN OF CUSTODY RECORD
0000—USATHAMA Installation

RESULTS DUE TO CUSTOMER: _____

SAMPLE EXTRACTION DATE: _____

SAMPLE ANALYSIS DATE: _____

PROJECT; JOB/TASK	LOT	ANALYSIS

Date Sampled	Site ID	DCL Sample Number(s)	Sample Matrix	Depth (ft)	Field Sample Number	Moisture Lot	Remarks

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)

Final Disposition: _____ Signature: _____